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INSTALLATION AND USE OF AN OXYGEN ANALYSER FOR THE 20-L EXPLOSION VESSEL

K.J. JUDGE

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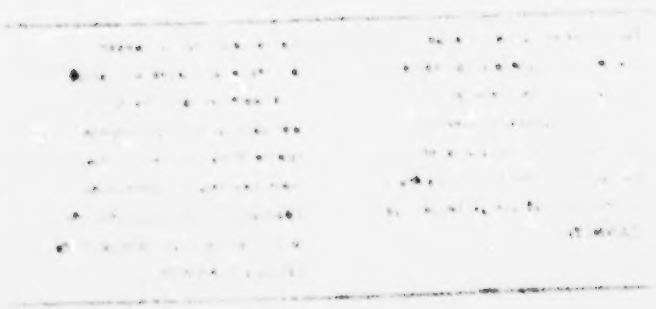

INSTALLATION AND USE OF AN OXYGEN ANALYSER FOR THE 20-L VESSEL

by

K.J. Judge*

ABSTRACT

Explosions involve exothermic reactions and form various products which often include oxides. To understand an explosion it is often useful to know to what extent the reactions involved have taken place. The consumption of oxygen can be used to determine this extent in explosions where it is a reactant. Coal and sulphide ore dust explosions consume oxygen and produce poisonous sulphur dioxide. Such reactions are studied at the Canadian Centre for Mineral and Energy Technology (CANMET).

An oxygen analyzer has been installed in the dust explosion lab at CANMET to assist in dust explosion research. Equipment modification, set up, calibration and operation are detailed.

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KEYWORDS: Oxygen analyzer; 20-litre vessel; dust explosions

INSTALLATION ET UTILISATION D'UN APPAREIL D'ANALYSE DE L'OXYGENE POUR LE RECIPIENT DE 20 LITRES

par

K.J. Judge*

RESUME

Une explosion est une réaction exothermique dont les divers produits comportent souvent des oxydes. Pour comprendre ce qu'est une explosion, il est souvent utile de savoir dans quelle mesure les réactions en jeu ont ou lieu. La consommation d'oxygène qui est un des agents qui interviennent dans une explosion peut être considérée comme une telle mesure. Les explosions de poussière de charbon et de minerai sulfuré consomment de l'oxyène et produisent du dioxyde de soufre toxique. De telles réactions sont étudiées au Centre canadien de la technologie de l'énergie et des minéraux (CANMET).

Un analyseur d'oxyène a été installé au laboratoire de recherche sur les explosions de poussière de CANMET pour appuyer la recherche sur les explosions de poussière. La modification, le montage, l'étalonnage et l'exploitation du matériel sont détaillés.

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MOTS CLES: analyseur d'oxyène; récipient de 20 litres; explosions de poussière

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INTRODUCTION

This report details the theory, installation, modification and calibration of the oxygen analysis equipment used at the Canadian Explosive Atmospheres Laboratory (CEAL). An oxygen analyzer was installed to provide a means of determining the consumption of oxygen during explosion tests carried out in the 20-L vessel. Also, measurement of the initial oxygen concentration can be used in tests to determine the minimum oxygen concentration for explosion of a given material. Included are step-by-step instructions for the use and calibration of the equipment as well as a correction curve for day to day use.

In general, the explosion testing carried out in the 20-L vessel at CEAL involves the dispersion of a dust in an evacuated chamber using a known gas or gas mix. Usually, the gas mix is compressed air which contains 21% oxygen. Sometimes, other "atmospheres" containing different amounts of oxygen may be used. In any case, the difference between the initial and final oxygen concentrations may be used as an indicator of the extent to which oxidation reactions have occurred during an explosion.

THEORY OF OPERATION

Oxygen is a paramagnetic gas and is, therefore, attracted by magnetic fields. The susceptibility of oxygen to a magnetic field is much greater than that of other common gases. The only other common gases that are paramagnetic, i.e. can interfere, are nitric oxide and nitrogen dioxide. The latter is, however, only weakly paramagnetic. Most other gases are diamagnetic and are thus repelled by a magnetic field. In quantitative terms the diamagnetism is sufficiently small that it can be neglected relative to paramagnetism. This paramagnetism is used in analyzers to measure oxygen.

A diamagnetic body (shaped like a dumb-bell) is suspended in a strong non-uniform magnetic field. An increase in the strength of the magnetic field will rotate the dumb-bell away from the field. This increase can be attained by the introduction of oxygen. The induced rotation is detected optically using a beam of light directed at a mirror which is mounted at the center of the dumb-bell. Optical sensors mounted on either side of the reflected beam provide a feedback signal which is used to charge the dumb-bell, via a conductive suspension filament, thereby re-centering the dumb-bell. The output from such an analyzer is linear and quite reliable.

Some factors can, however, affect the readings of the analyzer. Since the deflection of the dumb-bell is proportional to the concentration of oxygen in the area of the sensor and not the percent of oxygen in the gas, the effect of pressure must be

considered. For example, if the pressure is doubled for a given gas, then, assuming the ideal gas law, the analyzer would indicate twice the actual percentage as there would be twice as much oxygen in the test cell.

Normal fluctuations in atmospheric pressure are less than 2%. At most, this would cause a 2% error in oxygen reading i.e. 0.2% for a 10% oxygen mix or 2% on a 100% or pure oxygen sample. Calibrations should therefore, be carried out under the same pressure conditions as the sample under test, i.e. the analyzer vented to atmospheric pressure. If zero and span checks are performed regularly, the effective error from pressure change should be limited to at most 1% which is generally smaller than the range of repeatability of the explosions.

Similarly, temperature change has an effect on the magnetic properties of gases. An increase in temperature will increase the magnetic susceptibility of a gas resulting in an apparently greater percentage reading. Temperature also causes an expansion of gases which has the opposite effect. Therefore, the sensor and the gas temperature should be controlled.

Flow rate also can affect the reading by causing turbulence if the rate is too high, or slow response if it is too low. Thus the air flow must be controlled within certain limits.

APPARATUS

The apparatus chosen consists of a modified Beckman oxygen concentration analyzer (Model F3), a Matheson flowmeter (Model R7641), an ADI Mini Dia-Vac diaphragm pump (Model M1931 OCT), glass wool particle filter and associated valves and tubing (Fig. 1)

The Beckman Model F3 oxygen concentration analyzer uses the dynamic servo-magnetic principle mentioned above(1). The Model F3 analyzer had an analog meter with 0-5% and 0-25% ranges. While the 0-25% range is suitable for many of our tests, often we wish to use an enriched atmosphere of up to 100% oxygen. Therefore, we would prefer the analyzer to operate over the 0 to 100% range. Also, a digital output is preferred to the analog one provided.

A flowmeter (Matheson 7641 Rotameter with 601 tube and float) was purchased and is located just upstream of the oxygen analyzer. A setting of glass ball = 100 gives a flow rate of about 85 cubic centimeters per minute which is suitable for the analyzer and eliminates any errors which might result from varying flow rates.

Often, upon cooling, the pressure in the 20-L vessel will be below atmospheric pressure and the combustion gases must be pumped from the vessel through the analyzer. A vacuum pump (ADI Mini Dia-Vac single stage diaphragm pump) was purchased for this purpose. This pump has sufficient capacity to pump the required 85 cubic centimeters per minute at the typical post

combustion pressures in the 20-L vessel for two to three minutes (the time required to purge the lines and analyzer and give a steady reading).

A simple in-line glass wool particle filter prevents dust and combustion particles from entering the oxygen analyzer, flow meter or vacuum pump. This filter is located between the vacuum pump and the 20-L vessel sampling port.

MODIFICATION OF APPARATUS

A digital readout has been mounted on top of the oxygen analyzer and replaces the analogue display. This meter is calibrated to give a direct readout in percent oxygen (see "CALIBRATION"). The old display was a 1250 ohm 200 microamp current meter while the new meter is a 0-2.000 V voltmeter (Electro Numerics Model B35-P87). The old meter was replaced with a 1.2K ohm precision resistor and the new meter was connected across this resistor, and R19 (Appendix 1). A 100K ohm multiturn trim potentiometer was connected across the negative input and the tie point of the meter. The potentiometer wiper was connected to the positive input. By adjusting the potentiometer, the analyzer output was scaled to the meter input giving 0.500 V for a 50.0% oxygen concentration. This pot should not be used for daily calibration checks. Instead, the "SPAN" pot on the front of the analyzer should be used.

A calibration was carried out on the analyzer using a Stanton-Redcroft Critical Oxygen Index Equipment as the mixing

apparatus and standard. The purpose was to see if the F3 analyzer responded linearly and if the amplifier circuitry would operate up to 100% oxygen. Figure 2 is a plot of this data and shows a good linear correspondence up to 72% (for data see Appendix 2). After this point the output no longer responded to an increase in concentration. This lack of response suggested that either the dumb-bell was being mechanically restricted or that the amplifier's output voltage swing was limited to a level equivalent to 72% oxygen. This output voltage swing was found to be -.3v to 55 V. The 55 V output corresponds to a 72% oxygen reading and is, therefore, the limiting factor. Work will be performed to design and test a circuit which should extend the voltage swing to 74 V dc (55 * 100 / 72) sufficient to reach a 100% reading. The dc regulators provide 108 V which should allow the required output.

INSTALLATION

A regulating valve (Whitey) in the sampling port on the 20-L vessel is used to control the flow of combustion gases to the oxygen analyzer (Fig. 3). Glass wool loosely packed in a glass tube acts as an in-line particle filter protecting the vacuum pump, flow meter and analyzer from material which may obstruct proper flow or in time affect the calibration of the meter or operation of the pump. The vacuum pump draws the combusted gases through the in-line filter and pumps it to the analyzer distribution panel, flowmeter and analyzer. The panel has four connections labeled "ZERO", "SPAN", "SAMPLE" and "AUX".

One of these four inlets is selected by the 5-way ball valve and the gas is passed on to the regulating valve which controls the flow to about 85 cubic centimeters per minute (when the glass ball is set at 100).

The "ZERO" and "SPAN" inlets are used for calibrating the analyzer. "ZERO" gas (100% nitrogen) is used to set the zero reading and "SPAN" gas (an accurately analyzed gas mixture; nominally 50% oxygen in nitrogen) is used to adjust the gain to give a direct reading in percent oxygen from the analyzer. The "SAMPLE" inlet is used to connect the outlet of the vacuum pump through the selector valve to the analyzer via the flow meter. Considerations must be given to the partial pressure effect on the oxygen reading compared to the oxygen content of the 20-L vessel. This is discussed further under "Calculations". The fourth inlet, marked "AUX", may be used to vent the analyzer or to sample a fourth gas. This fourth gas may be a check on the gas being used in the pressurization tank (e.g. extra dry air nominal 21% oxygen).

The temperature inside the analyzer is controlled by a heater and maintained above room temperature. The manual recommends inlet gas temperatures be in the range 10-433C (50-1103F). Cooling of the gas through expansion has proven not to bring the temperatures below 103C and would not appear to be of any significance. Heat released during most explosion tests does warm the vessel, but certainly not above 433C. For more energetic reactions such as aluminum the vessel may feel hot, in which case, the gas should be allowed to cool before

passing it through the analyzer.

CALIBRATION

The modified apparatus was calibrated against the Stanton-Redcroft Critical Oxygen Index Apparatus. A "tee" fitting and shut off valve were installed in this index apparatus to allow its use as an analyzer/mixer. The installed outlet from the Stanton-Redcroft was connected to the "SAMPLE" inlet on the Beckman. The Stanton-Redcroft was set up as per the instructions except that nominal 50% (actually 48.9%) oxygen in nitrogen was used as the set point instead of pure oxygen. This was done to give a more accurate calibration over the range of the Beckman (0-72%) and to use the same check points (0 and 50%) on both analyzers. Using extra dry nitrogen and the standard (48.9% oxygen in nitrogen at the time of writing) the "ZERO" and "SPAN" of the two analyzers were adjusted to give the appropriate readings. The standard was replaced with 100% oxygen to allow mixes up to 72% and a calibration was performed.

The data points were taken in random order and to cover the 0 to 72% range in 5% increments. Also, included in the data are the set points and extra dry air. The data may be found in Appendix 3 and are presented in Figures 4 and 5. Figure 4 shows the correlation between the two analyzers while Figure 5 gives the corrections to be added to the Beckman reading to obtain the actual reading. This correction assumes the Stanton-Redcroft readings to be accurate. This assumption appears to be valid

since the reading for extra dry air was 21.0% which is within reading error (¯0.1) of its nominal concentration (20.9%) i.e. the Stanton-Redcroft is very linear in the range 0 to 50%.

PRELIMINARY TESTS AND CALCULATIONS

Two trials were carried out to determine the amount of oxygen used in a reaction. These trials were performed on different concentrations of lycopodium powder. The first trial resulted in a weak explosion and should have consumed little oxygen. The second trial, at twice the concentration, produced a more vigorous explosion and should have consumed considerably more oxygen. Figures 6 and 7 show the "pressure curves" for these two explosions, an empty vessel trial with just an "ignitor" and the "reaction pressure" which is the difference between the two. The data for these trials are reported in Appendix 5.

The amount of oxygen in the 20-L vessel can be calculated as follows:

$$n = PVc / RT$$

where P = pressure in atmospheres

n = number of moles

V = volume (in litres)

c = percentage (by volume) of O_2 in mix

R = gas constant (0.08205 L-atm / deg-mole)

T = absolute temp. (3C + 273)

For the test trials, the initial amounts of oxygen were the same (within experimental reproducibility) since the 20-L vessel was evacuated and then pressurized with "extra dry" air from the compression vessel in the same manner for each test. The air solenoid valve is opened for a period timed to result in a pressure of 1 atmosphere at the moment of ignition. While the pressure in the vessel, at the moment of ignition, is one atmosphere it is not at equilibrium due to the rapid expansion and dispersion of the gas. This matter is discussed in detail elsewhere (3). An empty vessel trial was performed to observe the final equilibrium temperature and pressure. The room temperature was 203C and the pressure reading was 0.662 V.

Using: - 101.3 kPa / atmoshpere

- 136 kPa / volt pressure calibration factor

the initial amount of oxygen was then calculated to be:

$$O_2 \text{ init.} = \frac{PVc}{RT} = \frac{(0.662 \times 136) \times 20 \times 0.213}{101.3 \times 0.08205 \times 293}$$

$$= 0.662 \times 136 / 101.3 \quad \text{atm.}$$
$$\times 20 \quad \text{litres}$$
$$\times 21.3\% \quad \%\ O_2 \text{ corrected}$$
$$\times 1 / 0.08205 \quad \text{deg-mole / L-atm}$$
$$\times 1 / [20 + 273] \quad 1 / 3K$$
$$= 0.157 \quad \text{moles}$$

The ignitor did not consume a noticeable amount of oxygen and after cooling the pressure was the same as without an ignitor.

For the two trials the final amount of oxygen was:

$$\text{Trial 1: } O_2 \text{ final} = \frac{PVc}{RT} = \frac{(0.610 \times 136) \times 20 \times 0.158}{101.3 \times 0.08205 \times 294}$$

$$= 0.107$$

$$O_2 \text{ consumed Trial 1} = 0.157 - 0.107$$
$$= 0.050 \text{ moles}$$

$$\text{Trial 2: } O_2 \text{ final} = \frac{PVc}{RT} = \frac{(0.588 \times 136) \times 20 \times 0.120}{101.3 \times 0.08205 \times 294}$$

$$= 0.079$$

$$O_2 \text{ consumed Trial 2} = 0.157 - 0.079$$
$$= 0.078 \text{ moles}$$

CONCLUSIONS

As shown, data from the oxygen analyzer can be used to calculate the amount of oxygen consumed in an explosion. In the preliminary trials the first explosion, which was weak, used less oxygen than the second explosion, which was stronger. The oxygen consumed did act as an indication of the extent to which the reactions took place and can readily be measured using this apparatus.

The oxygen analyzer system developed here as an easy-to-use device will be a useful routine tool to supplement the existing pressure-time instrumentation and further our knowledge of dust explosions.

ACKNOWLEDGEMENTS

The writer wishes to thank Dr. K.J. Mintz for his guidance and understanding during this installation period.

REFERENCES

1. Beckman Instruments, Inc., "Instruction Manual, Model F3 Oxygen Analyzer", Bulletin 1040-A.

2. Mintz, K.J., "Procedure for dust explosion tests using the 20 L vessel", Div. Rep. MRL 88-79(TR).

3. Mintz, K.J., "Commissioning of the 20-L vessel for studying dust-air explosions", Div. Rep. MRL 88-32(TR).

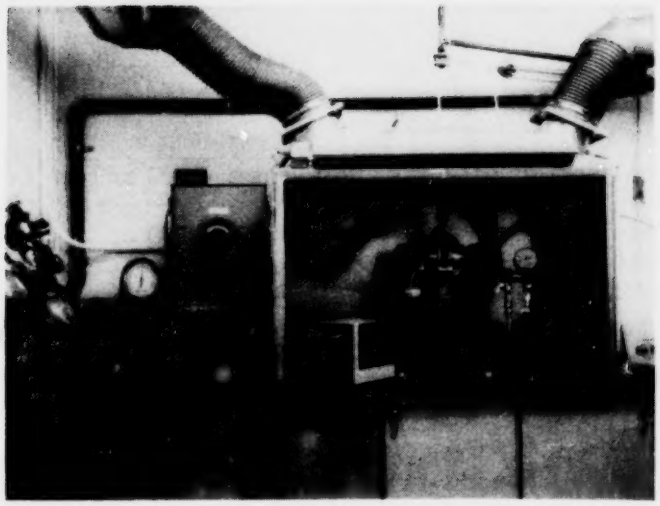
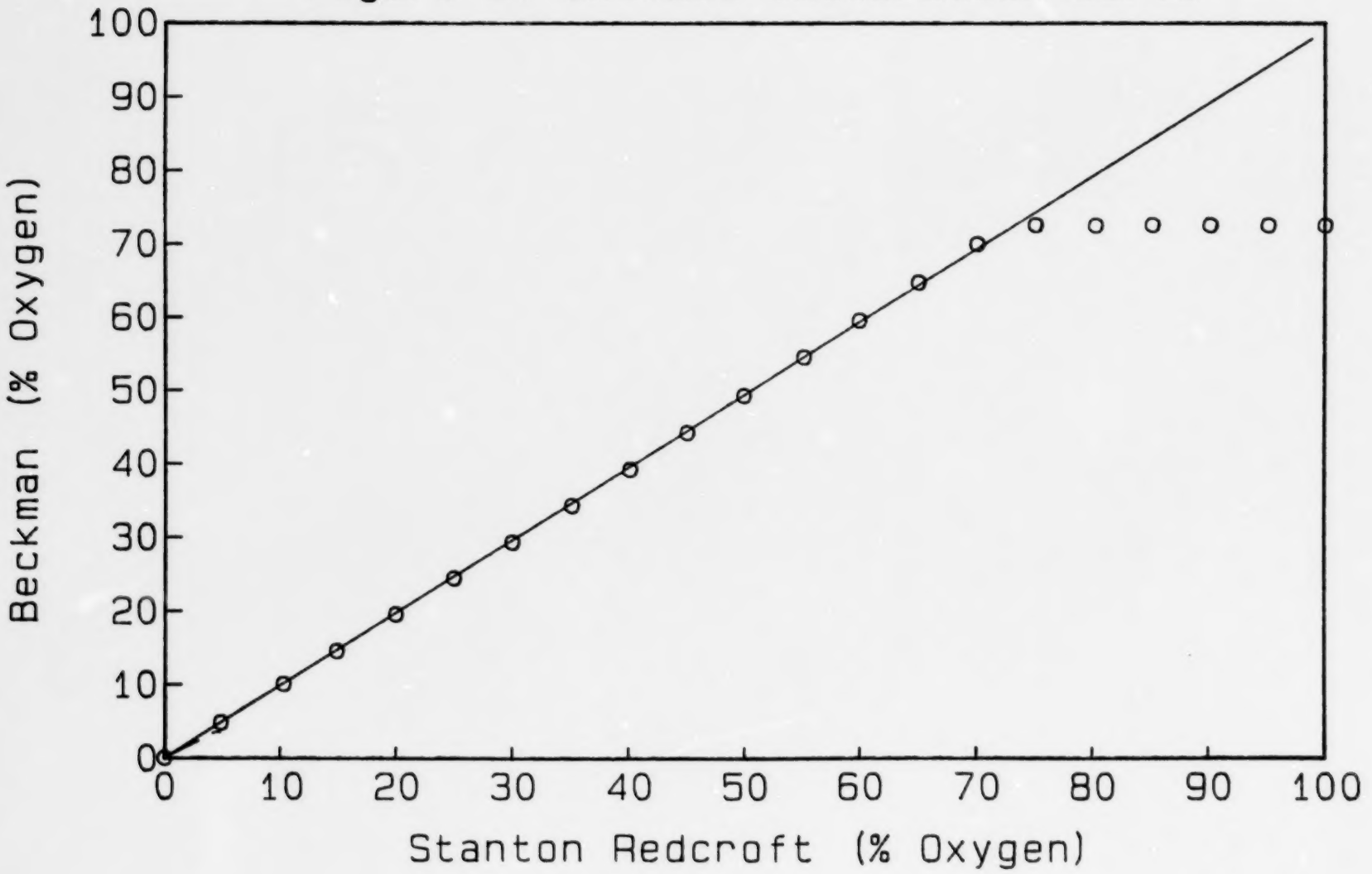


Figure 1 Photo of system equipment

Figure 2. Initial calibration curve



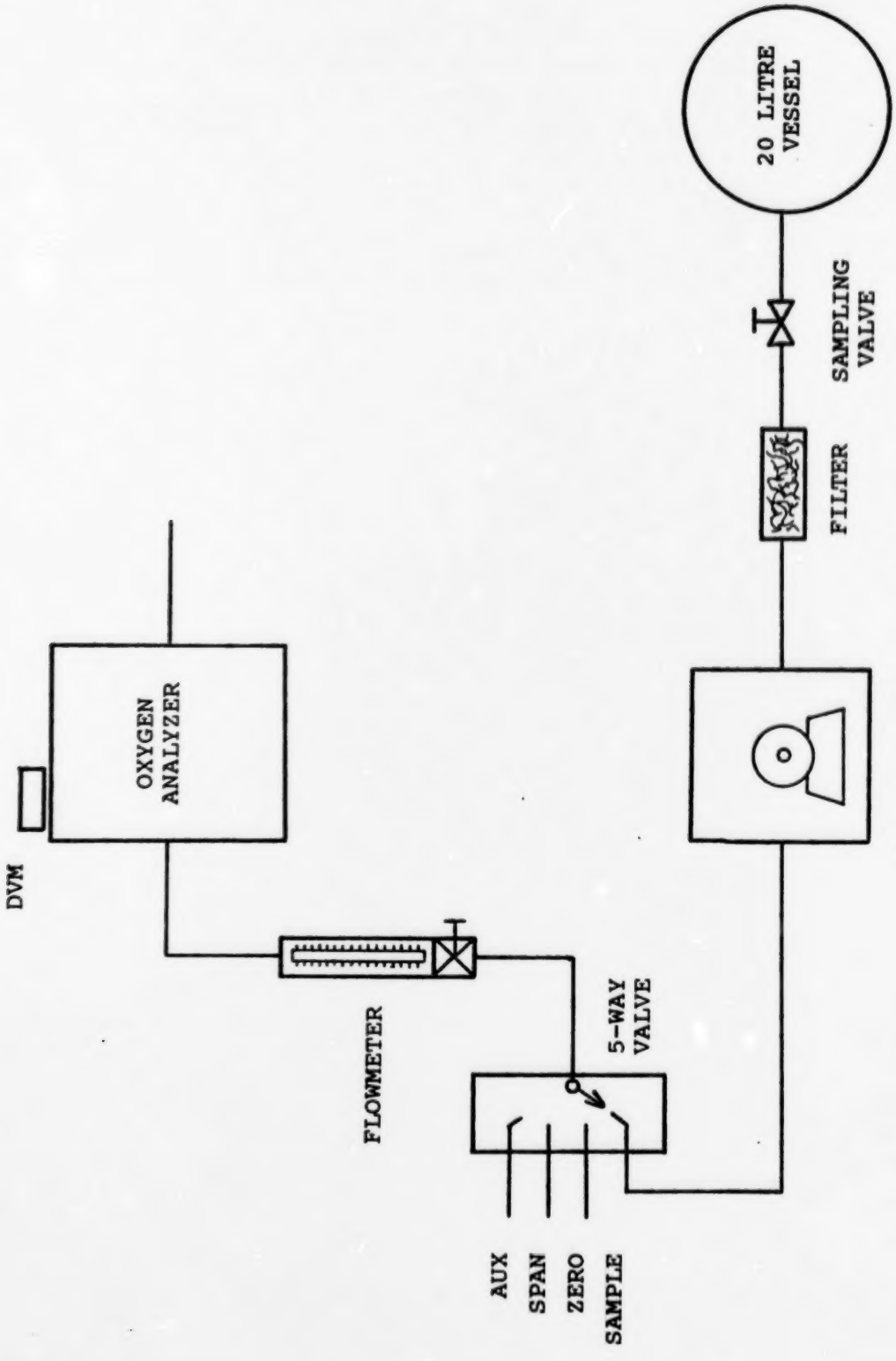


Figure 3. System block diagram

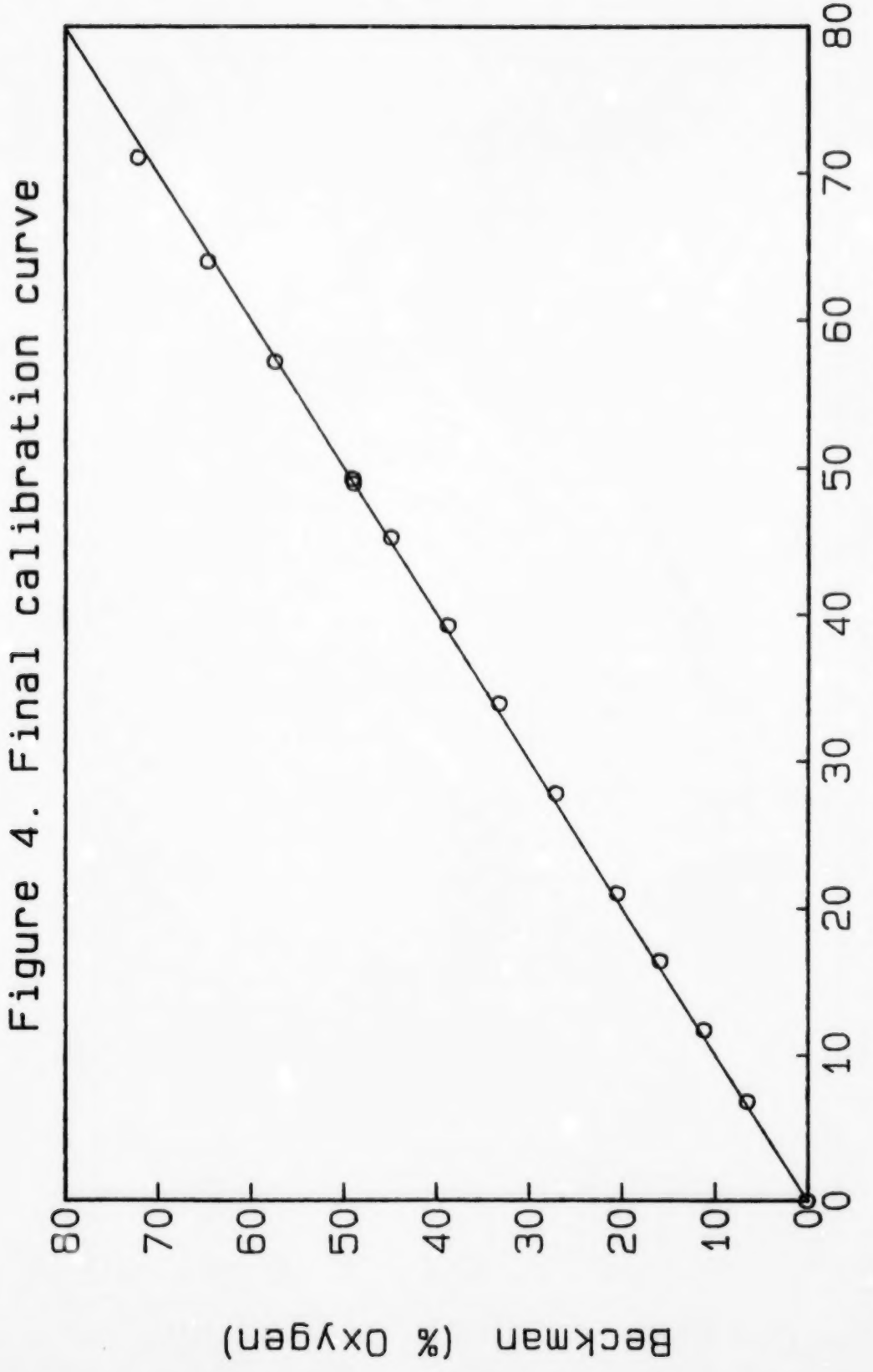
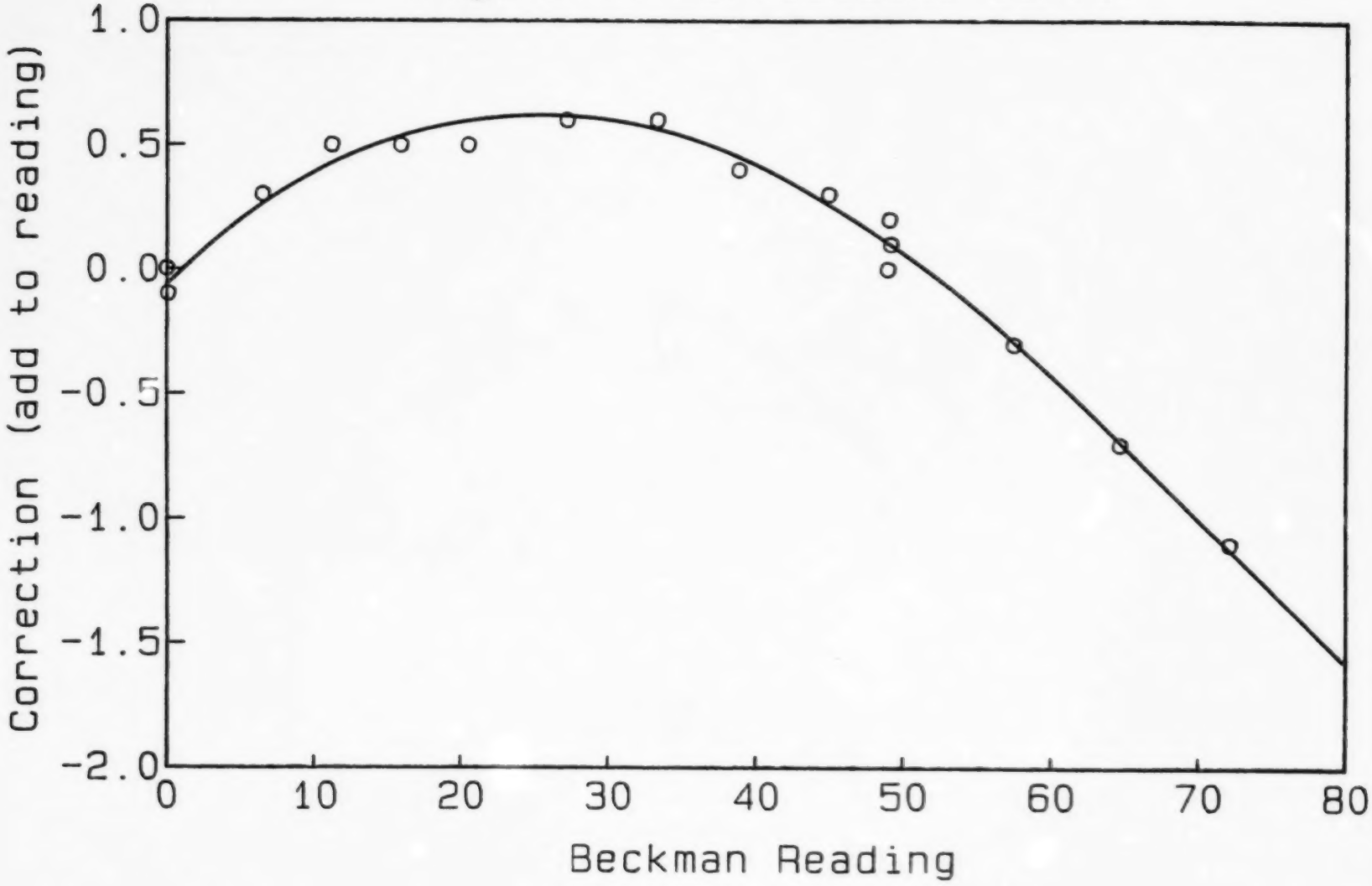


Figure 4. Final calibration curve
Beckman (% Oxygen)
Stanton Redcroft (% Oxygen)
0
10
20
30
40
50
60
70
80

Figure 5. Corrections curve



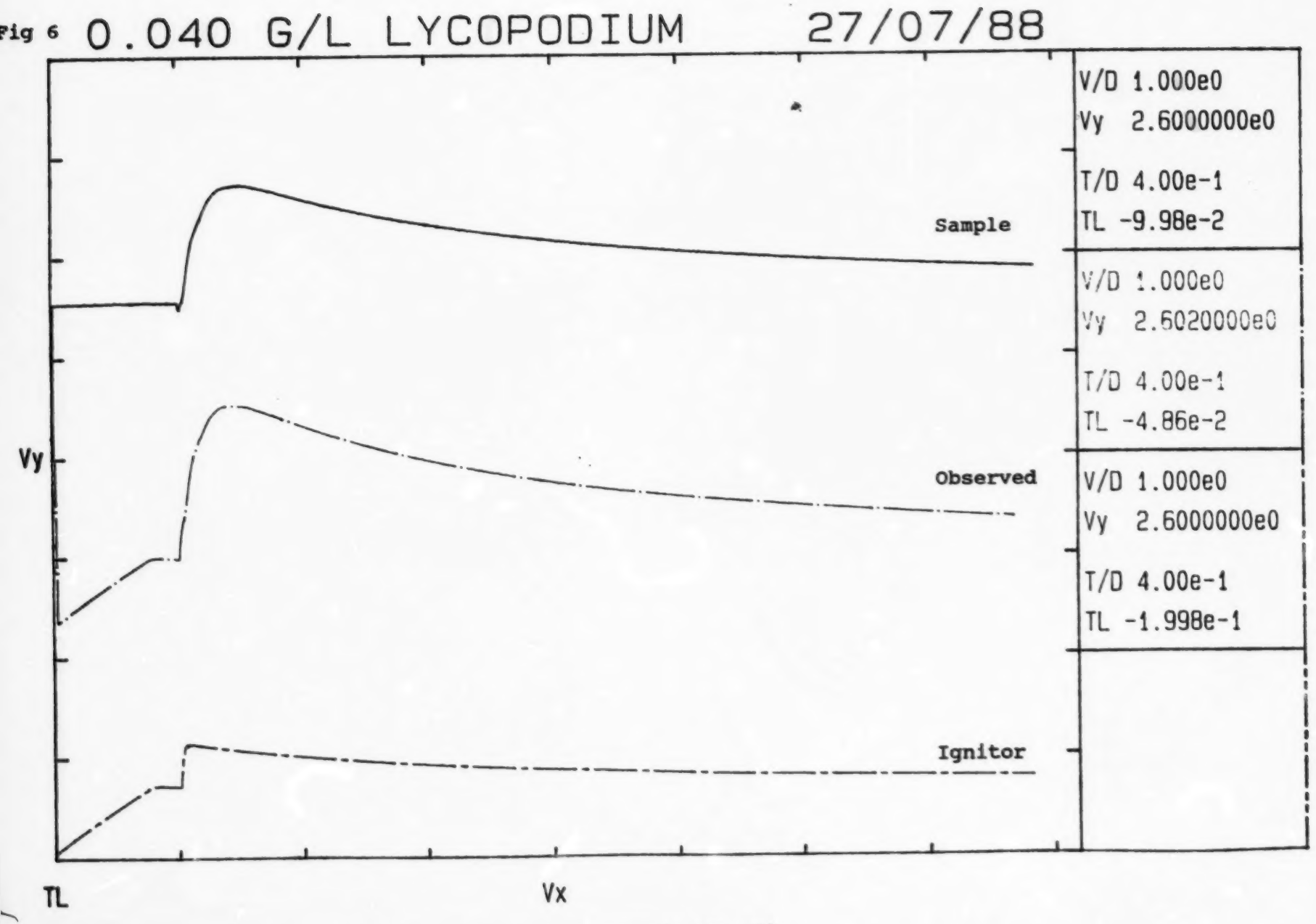


Fig. 6 - Lycopodium pressure curve (0.040 g/l).

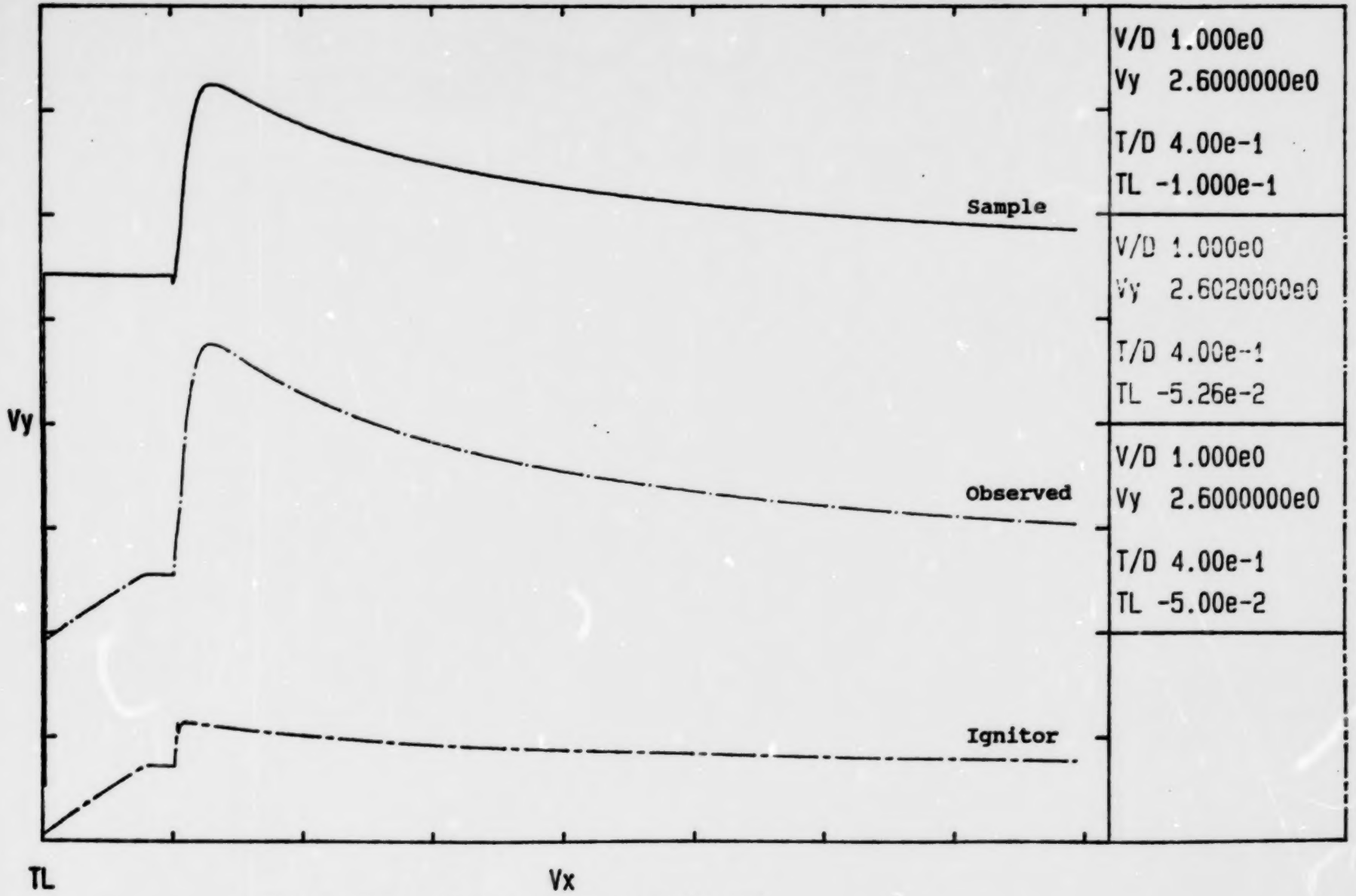
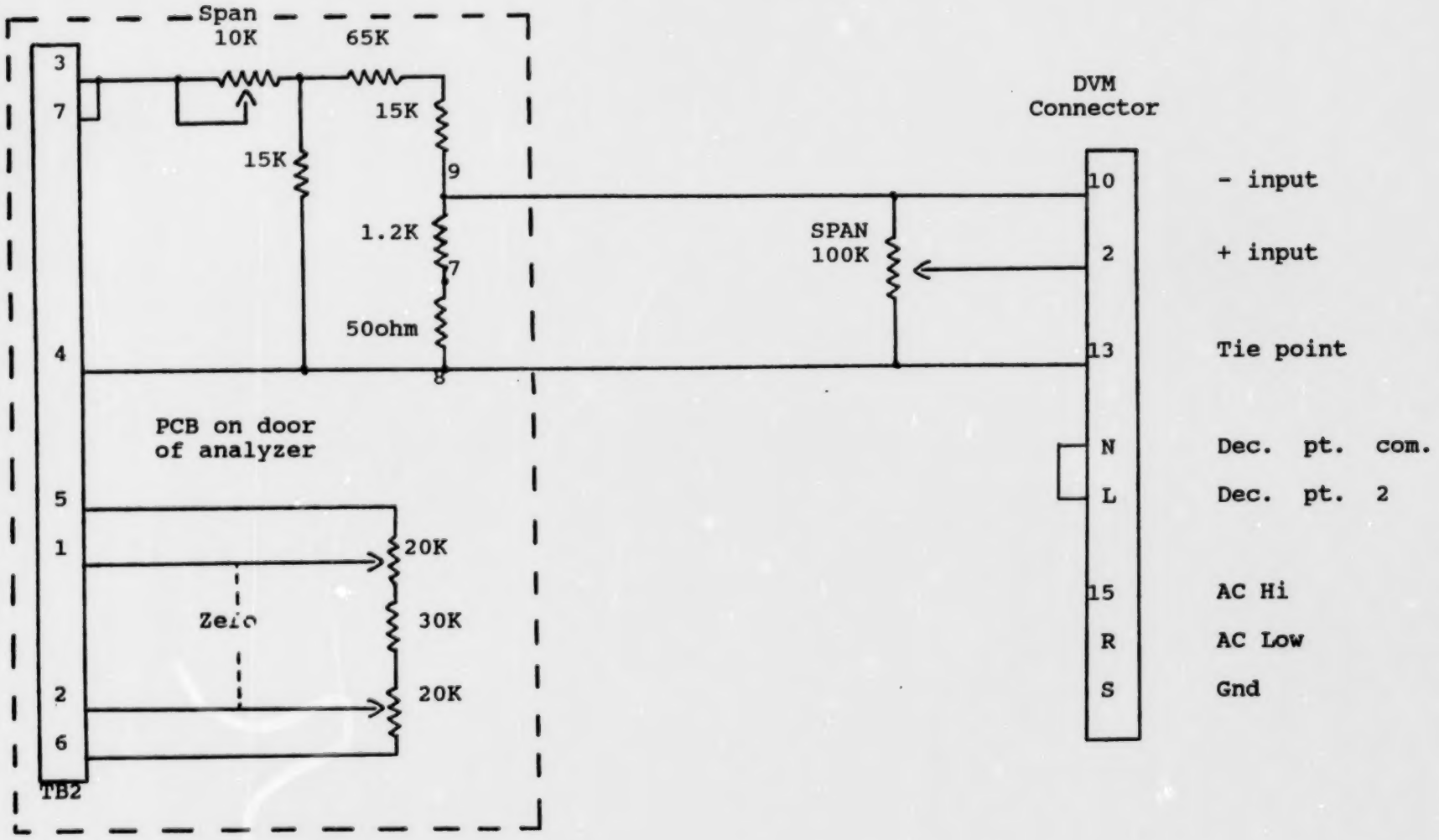


Fig. 7 - Lycopodium pressure curve (0.083 g/l).

APPENDIX 1

Schematic diagram (modifications)



APPENDIX 2

HP-plot

```
title:Figure 2. Initial calibration curve
X-axis:          Stanton Redcroft (% Oxygen)
   range:       .0000 to    100.0000
X-labels:0         10         20         30         40         50
60         70         80         90         100


Y-axis:          Beckman (% Oxygen)
   range:       .0000 to    100.0000
Y-labels:0         10         20         30         40         50
60         70         80         90         100


Graphical instructions read from file: initialcal
Data read from file:      initialdat


 set  point    x-value     y-value

    1     1      .0000       .0000
    1     2     4.9000      4.8000
    1     3    10.3000     10.1000
    1     4    14.9000     14.6000
    1     5    20.0000     19.6000
    1     6    25.0000     24.5000
    1     7    30.0000     29.4000
    1     8    35.1000     34.4000
    1     9    40.1000     39.3000
    1    10    45.0000     44.3000
    1    11    49.9000     49.3000
    1    12    55.1000     54.5000
    1    13    59.9000     59.5000
    1    14    65.0000     64.7000
    1    15    70.0000     69.9000
    2     1    75.0000     72.5000
    2     2    80.2000     72.4000
    2     3    85.1000     72.5000
    2     4    90.1000     72.5000
    2     5    95.1000     72.5000
    2     6   100.0000     72.5000
 Fitted line is: y =     .0000000 +     .9902633 x
standard deviation of slope =     .0017607
coefficient of correlation,r, =  .999945
line is fixed to pass through point       .000,      .000

 Fitted line is: y =     .0000000 +     .8198419 x
standard deviation of slope =     .0356134
coefficient of correlation,r, =  .387334
line is fixed to pass through point       .000,      .000
```

APPENDIX 3

HP-plot

```
title:Figure 4. Final calibration curve
X-axis:          Stanton Redcroft (% Oxygen)
   range:       .0000 to     80.0000
X-labels:0         10         20         30         40         50
60         70         80


Y-axis:          Beckman (% Oxygen)
   range:       .0000 to     80.0000
Y-labels:0         10         20         30         40         50
60         70         80


Graphical instructions stored in file: finalcal
 Data stored in file:      finaldata

 set  point    x-value     y-value

    1     1     48.9000     48.9000
    1     2       .0000       .0000
    1     3      6.8000      6.5000
    1     4     11.7000     11.2000
    1     5     16.4000     15.9000
    1     6     27.8000     27.2000
    1     7     33.9000     33.3000
    1     8     39.2000     38.8000
    1     9     45.2000     44.9000
    1    10     49.2000     49.1000
    1    11     57.1000     57.4000
    1    12     63.9000     64.6000
    1    13     71.0000     72.1000
    1    14       .0000       .1000
    1    15     49.2000     49.0000
    1    16     21.0000     20.5000
 Fitted line is: y =      .0000000 +    1.0012440 x
standard deviation of slope =      .0030472
coefficient of correlation,r, =  .999852
line is fixed to pass through point       .000,       .000
```

APPENDIX 4

HP-plot

```
title:Figure 5. Corrections curve
X-axis:          Beckman Reading
   range:       .0000 to     80.0000
X-labels:0         10         20         30         40         50
60         70         80


Y-axis:          Correction (add to reading)
   range:     -2.0000 to      1.0000
Y-labels:-2.0      -1.5       -1.0       -0.5       0.0        0.5
1.0


Graphical instructions read from file: correct
Data read from file:      correction


 set  point    x-value     y-value

    1     1     48.9000       .0000
    1     2       .0000       .0000
    1     3      6.5000       .3000
    1     4     11.2000       .5000
    1     5     15.9000       .5000
    1     6     27.2000       .6000
    1     7     33.3000       .6000
    1     8     38.8000       .4000
    1     9     44.9000       .3000
    1    10     49.1000       .1000
    1    11     57.4000      -.3000
    1    12     64.6000      -.7000
    1    13     72.1000     -1.1000
    1    14       .1000      -.1000
    1    15     49.0000       .2000
    1    16     20.5000       .5000
 Fitted line is: y =      .0000000 +     -.0013807 x
standard deviation of slope =      .0030379
coefficient of correlation,r, = -.585532
line is fixed to pass through point       .000,       .000
```

APPENDIX 5

PRELIMINARY TRIALS DATA

	Blank	Trial #1	Trial #2
Sample	none	lycopodium	lycopodium
Mass (mg)	----	0.8006	1.6729
Conc. (g/l)	----	0.0400	0.0836
Disk	Ideal Metals - June 1988		
Track	8	9	10
Cal. factor (KPa/V)	136	136	136
Pres. max. (KPa)	---	208	300
t_m (ms)	---	173	116
$(dP/dt)_m$ (KPa/s)	---	3060	5423
t_r (ms)	---	26.4	26.0
P_f (mv)	662	610	588
P_f (KPa)	84.3	83.0	80.0
T_{room} (°C)	20	21	21
O_2 reading (%)	20.7	15.3	11.6
correction (%)	+0.6	+0.5	+0.4
O_2 corrected (%)	21.3	15.8	12.0
Moles final	0.172	0.117	0.086
Moles consumed	----	0.055	0.086

APPENDIX 6

SAMPLING PROCEDURE FOR OXYGEN ANALYZER

1. Plug in oxygen analyzer at least 24 hours in advance of test.

2. Connect nitrogen and 50% oxygen bottles to their respective inlets on the analyzer plumbing panel.

3. Open the shut-off valves on the nitrogen and 50% oxygen bottles.

4. Adjust the regulators on the nitrogen and 50% oxygen bottles to around 70kPa (10 psi) and open the outlet valves.

5. Set the source selector knob on analyzer plumbing panel to "ZERO".

6. Adjust the gas flow via the valve on the flow meter to:
   glass ball = 100

7. Wait about two minutes for a steady reading and adjust the "ZERO" control on analyzer so that the digital readout indicates 00.0.

8. Set the source selector knob to "SPAN".

9. Adjust the flow if necessary as in 6.

10. Wait about two minutes for a steady reading and adjust the "SPAN" control on the analyzer so that the digital readout indicates the analyzed value (nominally 50%).

11. Repeat steps 4 - 9 until the zero and span repeat to within +/-0.1.

12. Set the source selector knob to "SAMPLE">

13. Close the sampling outlet (Whitey) valve on the 20-L vessel.

14. Perform explosion test in the normal manner (2).

15. If the 20-L vessel is warm, wait for it to cool and the pressure to stabilize.

16. Record the pressure from the Nicolet:
    reading multiplied by calibration factor
    calibration factor at time of writing = 136 KPa/volt

18. Turn on the sampling pump.

19. Connect the sampling outlet (Whitey valve) from the 20 L vessel to the the inlet of the sampling pump.

20. Ensure that the inline dust filter is in position between the 20-L vessel and the pump and that the glass wool is clean.

21. Connect the sampling pump outlet to the analyzer plumbing panel inlet marked "SAMPLE".

22. Close the needle valve on the flow meter, then open 1 turn.

23. Open the 20-L vessel sampling outlet (Whitey) valve.

24. Adjust the flow rate over the next 2 minutes using the flowmeter valve and wait until a stable reading is observed.

25. Record the reading.

26. Set the source selector switch to "ZERO".

27. Close the 20-L vessel sampling outlet (Whitey) valve.

28. Turn off the sampling pump.

29. Disconnect the sampling pump from the analyzer.

30. Disconnect the sampling pump from the 20-L vessel.

31. Wait for a stable reading and check the "ZERO".

32. Set the source selector switch to "SPAN".

33. Wait for a stable reading and check the "SPAN".

34. If the "ZERO" and "SPAN" are not acceptable the test must be repeated.

35. If another explosion test is to be carried out repeat from step 12.

36. Set selector switch to "SAMPLE".

37. Close the shut-off valves on the nitrogen and 50% oxygen bottles.